United States Patent 5,905,063 Tanabe, et al. May 18, 1999

Tanabe teaches "A remover solution composition for resist which comprises (a) a salt of hydrofluoric acid with a metal-free base, (b) a water-soluble organic solvent, and (c) water and optionally (d) an anticor rosive, and has a pH or 5 to 8."

Tanabe teaches in claim 3, "The remover solution composition for resist as claimed in claim 1, wherein component (a) is ammonium fluoride"

Tanabe teaches in claim 2, "The remover solution composition for resist as claimed in claim 1, wherein component (a) is a salt of hydrofluoric acid with at least one metal-free base selected from the group consisting of hydroxylamines; primary, secondary, or tertiary aliphatic alicyclic, aromatic and heterocyclic amines; ammonia water; and C.sub.1 to C.sub.4 lower alkyl quaternary ammonium salt groups"

Tanabe teaches the water-soluble solvent in claim 5, "a water-soluble organic solvent which is at least one solvent selected from the group consisting of dimethyl sulfoxide, 3,3-dimethyl-2-imidazolidinone, and diethylene glycol monobutyl ether"

Tanabe generally teaches the water-soluble solvent to be "Examples of the water-soluble organic solvents include sulfoxides, sulfones, amides such as N,N-dimethylacetamide, lactams such as N-methyl-2-pyrrolidon e, imidazolidinones, lactones, and polyhydric alcohols (e.g., ethylene glycol, ethylene glycol inonomethy lether, ethylene glycol monoethyl ether, ethylene glycol monobutyl ether, ethylene glycol monoethyl ether, ethylene glycol monobutyl ether, ethylene glycol, etc."

Tanabe teaches corrosion inhibitors in amounts ranging from 0.5 to 15% including triazole compounds such as benzotriazole, o-tolyltriazole, m-tolyltriazole, p-tolyltriazole, carboxybenzotriazole, 1-hydroxybenzotriazole, nitrobenzotriazole, and dihydroxypropylbenzotriazole. Among these, preferred is benzotriazole.

Tanabe teaches weight percentages of the individual components in the following ranges: "In the case where the composition of the present invention comprises components (a) to (d), it is desirable that the content of component (b) is from 0.2 to 8% by weight, preferably from 0.5 to 5% by weight; the content of component (b) is from 40 to 80% by weight, preferably from 55 to 75% by weight; the content of component (d) is from 0.5 to 15% by weight, preferably from 0.5 to 10% by weight; and the remainder is component (c). If the amount of each component is outside the range shown above, the composition is impaired in the remo vability of modified films and in noncorrosive properties."

```
6228829
6306812
6242406
6399557
4102823
6306817
6218351
6387862
6280527
5565136
5026933
5284989
5334332
5245072
3480556
3582401
-----classlist-----
510/176
134/40
134/38
510/506
510/499
430/331
510/206
510/365
510/421
510/417
510/202
510/435
510/175
510/505
510/424
510/426
510/437
134/42
510/434
134/2
510/432
510/212
134/3
510/280
134/13
510/269
510/258
510/402
510/255
430/329
510/264
510/401
510/501
252/364
430/325
252/387
216/48
252/793
430/309
430/256
252/396
510/164
510/413
510/188
510/204
438/906
510/493
430/258
438/745
510/407
510/422
508/208
510/500
134/41
510/504
```

-----keywords-----

fluoroboric acid ammonium fluoride alkaline ph ammonium fluoride tetramethyl ammonium fluoride tetraethyl amine sulfoxide sulfone amide lactone pyrrolidone imidazolidinone glycol glycol ether propylene glycol e thylene glycol nmp methylpyrrolidone pyrrolidone pyrrodlidione ammonium hydroxide benzotriazole vanillin hepes weak acid weak benzotriazole ammonium hydroxide hydroxide ethylene glycol propylene glycol propylene glycol ether pyrrolidone lactone amide tetraethyl tetramethyl ammonium tetramethyl alkaline a mmonium fluoride fluoroboric acid buffered fluoroboric sulfoxide sulfone imidazolidinone nmp aqueous acid ammonium amine glycol ether ethylene methylpyrrolidone pyrrodlidione vanillin hepes

----- 5185235 classes:1 430/331 1 134/40 1 134/42 1 216/48 1 430/309 1 430/325 1 430/329 1 510/176 1 510/202 1 510/204 1 510/212 1 510/504 score: 826

keywords: glycol;propylene glycol;ethylene glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;ethylen
e glycol;propylene glycol;propylene;tetramethyl ammonium;tetramethyl;aqueous;ammonium;glycol;ether;ethyle
ne;

United States Patent: 5,185,235 FULL TEXT FORMAT --> (1of1) United States Patent 5,185,235 , &nbsp et al. February 9, 1993 Remover solution for photoresist The remover solution of the invention comprises (A) from 35 to 80% by weight of an alcoholic solvent such as \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, (B) from 10 to 40% by weight of an organic solvent which is a halogenated hydrocarbon solvent, e.g., 1,2-dichlorobenzene and methylene chloride, an \*\*ether\*\* solvent, e.g., tetrahydrofuran, or an aromatic solvent, e.g., benzene and xylene, and (C) from 0.1 to 25% by weight of a quaternay \*\*ammonium\*\* compound such as \*\*tetramethyl ammonium \*\*hydroxide\*\* and trimethyl hydroxyethyl \*\*ammonium \*\*hydroxide\*\*. Different from conventional remover solutions which only can swell cured photoresist compositions, the inventive remover solution has a power to completely dissolve a cured photoresist laver sist which consists essentially of, in admixture: (A) an aliphatic alcoholic solvent selected from the group consisting of methyl alcohol, ethyl alcohol, propyl alcohol, 3-methyl-3-methoxy butyl alcohol, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\* in a weight fraction in the

\*\*ether\*\* and dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\* in a weight fraction in the
 range from 35% to 80%;
(B) an organic solvent selected from the group consisting of halogenated
 hydrocarbon solvents, unhydroxylated \*\*ether\*\* solvents selected from the
 group consisting of dioxane, tetrahydrofuran, diethylene \*\*glycol\*\* dimethyl
 \*\*ether\*\*, triethylene \*\*glycol\*\* dimethyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* dimethyl
 \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* diethyl \*\*ether\*\*, diethylene \*\*glycol\*\* diethyl \*\*ether\*\* and

diethylene \*\*glycol\*\* dibutyl \*\*ether\*\*, and unhalogenated aromatic solvents in a

----- 4592787 classes:1 134/38 1 510/176 1 510/413 1 510/421 1 510/422 1 510/435 score: 704

keywords: alkaline;ph;glycol;glycol ether;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol;propylene;glycol ether;alkaline;aqueous;acid;ammonium;glycol;ether;ethylene;

```
tants may be used. It is further
       taught that it may be desirable to add up to 10 percent of an **alkaline**
       agent.
In the many heretofore known stripping compositions for stripping
       photoresist, there are components which are toxic, corrosive, or are
       hazardous to use. Furthermore, many of these stripping compositions do not
       strip the photoresist in an expedient manner.
What is needed is a composition useful in stripping photoresist polymers
       from substrates which does not contain toxic reagents, does not contain
       corrosive agents, and does not contain reagents which are hazardous to
       use. Furthermore, what is needed is a composition which results in faster
       stripping of the photoresist polymer mask.
SUMMARY OF THE INVENTION
This invention is a composition suitable for stripping cross-linked
       photoresist polymer which comprises
 (a) a lower alkyl monoether of a **propylene **glycol**, or a mixture of lower
      alkyl monoethers of **propylene **glycol**;
(b) a
      t the
      photoresist polymer from the substrate, so as to break up the coating. Further, the lower alkyl monoethers of **propylene **glycol** serve as cosolvents for the photoresist polymer. Preferred lower alkyl monoethers
       of **propylene **glycol** correspond to the formula
       ##STR1##
       wherein
R.sup.1 is C.sub.1-4 alkyl;
R.sup.2 is hydrogen or methyl; and
n is an integer of 1 to 3,
with the proviso that one R.sup.2 in each unit is methyl and the other is
       hydrogen. Unit refers herein to the moiety
       ##STR2##
Examples of lower alkyl monoethers of **propylene **glycol** useful in this
       invention include **propylene **glycol** methylether, **propylene **glycol**
      **propylene **glycol** propylene **glycol** isopropylene **glycol** dipropylene **glycol** dipropylene **glycol** butylether, **propylene **glycol** isobutylether, dipropylene **glycol** methylether, dipropylene **glycol** ethylether, dipropylene **glycol**
      propylether, dipropylene **glycol** isopropylether, dipropylene **glycol**
       butylether, dipr
      opylene **glycol** isobutylether, tripropylene **glycol**
      methylether, tripropylene **glycol** ethylether, tripropylene **glycol**
propylether, tripropylene **glycol** isopropylether, tripropylene **glycol**
butylether and tripropylene **glycol** isobutylether. More preferred lower
alkyl monoethers of **propylene **glycol** include **propylene **glycol** methylether,

**propylene **glycol** ethylether, **propylene **glycol** isopropylether, **propylene**

**qlycol** propylether, dipropylene **glycol** methylether, dipropylene **glycol**

**glycol** propylether, dipropylene **glycol** methylether, dipropylene **glycol**
      propylether, dipropylene **glycol* methylether, dipropylene **glycol* propylether, dipropylene **glycol* isopropylether and tripropylene **glycol* methylether. Most preferred are **propylene **glycol** isopropylether and **propylene **glycol** methylether.
Any C.sub.2-6 alkanol is useful in this invention. The C.sub.2-6 alkanol
       functions as a penetrating solvent and is a good wetting agent for the
       substrate. Examples of C.sub.2-6 alkanols useful in this invention are
      ethanol, propanol, isopropanol, n-butanol, isobutanol, pentane and hexane.
      Pre
                   5968848
classes:1 438/745 1 134/2 1 134/3 1 134/41 1 438/906 1 510/176
keywords: ammonium fluoride; alkaline; ph; ammonium fluoride; glycol; propylene glycol; ethylene glycol; benzotr
iazole;benzotriazole;hydroxide;ethylene glycol;propylene glycol;propylene;alkaline;ammonium fluoride;fluo
ride; aqueous; acid; ammonium; amine; glycol; ether; ethylene;
      g a hydrofluorate remover solution, and is a composition
      containing **ethylene **glycol** and/or **propylene **glycol** and another
      water-soluble organic solvent.
The water-soluble organic solvent is a water-soluble organic solvent other
      than **ethylene **glycol** and **propylene **glycol**, and examples of such solvents
monohydric alcohols such as methyl alcohol, ethyl alcohol, and isopropyl
      alcohol:
```

```
ketones such as acetone;
sulfoxides such as dimethyl **sulfoxide**;
amides such as N,N-dimethylformamide, N-methylformamide,
     N, N-dimethylacetamide, N-methylacetamide, and N, N-diethylacetamide;
lactams such as N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone,
     N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, and
     N-hydroxyethyl-2-pyrrolidone;
imidazolidinones such as 1,3-dimethyl-2-imidazolidinone,
     1,3-diethyl-2-imidazolidinone, and 1,3-diisopropyl-2-
      butyric acid,
     isobutyric acid, oxalic acid, malonic acid, succinic acid, glutaric acid,
     maleic acid, fumaric acid, benzoic acid, phthalic acid, 1,2,3-benzenetricarboxylic acid, **glycol**ic acid, lactic acid, malic acid,
     citric acid, acetic anhydride, phthalic anhydride, maleic anhydride,
     succinic anhydride, and salicylic acid. Formic acid, phthalic acid,
     benzoic acid, phthalic anhydride, and salicylic acid are the preferred
     carboxyl-group-containing organic compounds. Phthalic anhydride and
     salicylic acid are especially preferred.
Examples of triazole compounds include **benzotriazole**, o-tolyltriazole,
    m-tolyltriazole, p-tolyltriazole, carboxybenzotriazole, 1-hydroxytriazole, nitrobenzotriazole, and dihydroxypropylbenzotriazole. Among these,
     **benzotriazole** is preferred.
Typical examples of saccharides include D-sorbitol, arabitol, mannitol,
     sucrose, and starch. Among these, D-sorbitol is preferred.
Each of the above-listed anticorr
     could not be observed,
     **ammonium **fluoride** salt was deposited during the rinse treatment.
                  TABLE 1
                           Degree of Corrosion
    Composition of
      Rinse solution
                                  after Dipping in Water
      (weight ratio)
                                  at 23.degree. C. for 20 min.
    Example 1
            Methanol:Ethylene **glycol**
                               Good
                       (50:50)
      Example 2 Methanol:Ethylene **glycol**
                                                             Good
                        (80:20)
      Example 3
                Ethanol:Ethylene **glycol**
                                                             Good
                        (70:30)
      Example 4 Ethanol:Ethylene **glycol**:
                                                             Good
                 Pyrocatechol (30:68:2)
      Example 5
                 Isopropyl Alcohol:Ethylene
                                                         Good
                 **glycol** (70:30)
      Example 6 Isopropyl Alcohol:Ethylene
                                                         Good
                 **glycol** (50:50)
      Example 7 Methanol: Propylene
              5905063
classes:1 510/176 1 134/13 1 134/2 1 134/3 1 134/40 1 134/42 1 510/255 1 510/258 1 510/264 1 510/269 1 51
0/401 1 510/402
score: 682
keywords: ammonium fluoride;ph;ammonium fluoride;sulfoxide;glycol;ethylene glycol;nmp;hydroxide;ethylene
glycol;ammonium fluoride;fluoride;buffered;sulfoxide;nmp;aqueous;acid;ammonium;amine;glycol;ether;ethylen
    lpiperidine, oxazole, and thiazole. Examples of the C.sub.1
    to C.sub.4 lower alkyl quaternary **ammonium** salt groups include
    **tetramethyl**ammonium **hydroxide** and trimethyl(2-hydroxyethyl)-ammonium
    **hydroxide** (choline). Preferred metal-free bases are ammonia water,
    monoethanolamine, and **tetramethyl**ammonium **hydroxide**, because these bases
    are easily available and safe. A salt of the metal-free base with
    hydrofluoric acid can be produced by adding the metal-free base to
    commercial hydrofluoric acid having a hydrogen **fluoride** concentration of
    50 to 60% in such a proportion as to result in a pH of 5 to 8. Commercial
```

\*\*ammonium \*\*fluoride\*\* is, of course, usable as the salt.
The composition of the present invention has an almost neutral pH value of

almost neutral. The proportion of hydrofluoric acid to a metal-free base to be added thereto so as to attain a neutral pH value va treatments, which are necessary in resist removing processes where hydrogen \*\*fluoride\*\* generates. Any organic solvent may be used as component (b) in the present invention, as long as it is miscible with components (a), (c), and (d). Water-soluble organic solvents used in conventional organic \*\*amine\*\*-based remover solution compositions may be used. Examples of the water-soluble organic solvents include \*\*sulfoxide\*\*s (e.g., dimethyl \*\*sulfoxide\*\*), \*\*sulfone\*\*s (e.g., dimethyl \*\*sulfoxide\*\*), \*\*sulfone\*\*, diethyl \*\*sulfone\*\*, bis(2-hydroxyethyl) \*\*sulfone\*, \*\*tetramethyl\*\*ene \*\*sulfone\*\*), \*\*amide\*\*s (e.g., N,N-dimethylformamide, N-methylformamide, N, N-dimethylacetamide, N-methylacetamide, N, N-diethylacetamide), lactams (e.g., N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, N-hydroxyethyl-2-pyrrolidone), \*\*imidazolidinone\*\*s (e.g., 1,3-dimethyl-2-imidazolidinone, 1,3-diethyl-2-imidazolidinone, 1,3-diisopropyl-2-imidazolidinone 1,3-disopropyl-2-imidazolidinone
), \*\*lactone\*\*s (e.g., .gamma.-butyrolactone,
.delta.-valerolactone), and polyhydric alcohols (e.g., \*\*ethylene \*\*glycol\*\*,
\*\*ethylene \*\*glycol\*\* inonomethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*,
\*\*ethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\*, monomethyl \*\*ether\*\*
acetate, \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\* acetate, diethylne \*\*glycol\*\*,
diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monoethyl \*\*ether\*\*,
diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*); and derivatives thereof. Among these, preferred are dimethyl \*\*sulfoxide\*\*, N,N-dimethylformamide, N, N-dimethylacetamide, N-methyl-2-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, ethlylene \*\*glycol\*\*, and diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, because these solvents bring about the removability of modified resist films. In particular, use of a water-soluble organic solvent comprising at least 10% by weight (wt %) \*\*ethylene \*\*glycol\*\* as component (b) is preferred in that the composition of the present invention contain 5792274 classes:1 134/13 1 134/2 1 134/3 1 134/40 1 134/42 1 510/176 1 510/255 1 510/258 1 510/264 1 510/269 1 51 0/401 1 510/402 score: 652 keywords: ammonium fluoride;ph;ammonium fluoride;sulfoxide;glycol;ethylene glycol;nmp;hydroxide;ethylene glycol; ammonium fluoride; fluoride; buffered; sulfoxide; nmp; aqueous; acid; ammonium; amine; glycol; ether; ethylen and dicyclohexylamine. Examples of the aromatic \*\*amine\*\*s include benzylamine, dibenzylamine, and N-methylbenzylamine. Examples of the heterocyclic \*\*amine\*\*s include pyrrole, pyrrolidine, \*\*pyrrolidone\*\*, pyridine, morpholine, pyrazine, piperidine, N-hydroxyethylpiperidine, oxazole, and thiazole. Examples of the C.sub.1 to C.sub.4 lower alkyl quaternary \*\*ammonium\*\* salt groups include \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* and trimethyl(2-hydroxyethyl)-ammonium \*\*hydroxide\*\* (choline). Preferred metal-free bases are ammonia water, monoethanolamine, and \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\*, because these bases are easily available and safe. A salt of the metal-free base with hydrofluoric acid can be produced by adding the metal-free base to commercial hydrofluoric acid having a hydrogen \*\*fluoride\*\* concentration of 50 to 60% in such a proportion as to result in a pH of 5 to 8. Commercial \*\*ammonium \*\*fluoride\*\* is, of course, usable as the salt. The composition of ms and inhibiting the composition from corroding a metal film on a substrate or peripheral devices including a remover feeder. Furthermore, a low hydrogen \*\*fluoride\*\* content of component (a) eliminates the necessity of troublesome exhaust and wastewater treatments, which are necessary in resist removing processes where hydrogen \*\*fluoride\*\* generates. Any organic solvent may be used as component (b) in the present invention, as long as it is miscible with components (a), (c), and (d). Water-soluble organic solvents used in conventional organic \*\*amine\*\*-based remover solution compositions may be used. Examples of the water-soluble organic solvents include \*\*sulfoxide\*\*s (e.g., dimethyl \*\*sulfoxide\*\*), \*\*sulfone\*\*s (e.g., dimethyl \*\*sulfone\*\*, diethyl \*\*sulfone\*\*, bis(2-hydroxyethyl) \*\*sulfone\*\*, \*\*tetramethyl\*\*ene \*\*sulfone\*\*), \*\*amide\*\*s (e.g., N,N-dimethylformamide, N-methylformamide, N,N-dimethylacetamide, N-methylacetamide, N,N-diethylacetamide), lactams

(e.g., N

report09881552.txt -methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, N-propyl-2-pyrrolidone, N-hydroxymethyl-2-pyrrolidone, N-hydroxyethyl-2-pyrrolidone), \*\*imidazolidinone\*\*s (e.g., 1,3-dimethyl-2-imidazolidinone, 1,3-diethyl-2-imidazolidinone, 1,3-diisopropyl-2-imidazolidinone), \*\*lactone\*\*s (e.g., .gamma.-butyrolactone, .delta.-valerolactone), and polyhydric alcohols (e.g., \*\*ethylene \*\*glycol\*\*, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*,

\*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\* acetate,

\*\*ethylene \*\*glycol\*\* monomethyl \*\*ether\*\* acetate, diethylene \*\*glycol\*\*, diethylene

\*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\*, diethylene

\*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*qlycol\*\* monobutyl \*\*ether\*\*); and derivatives thereof. Among these, preferred are dimethyl \*\*sulfoxide\*\*, N,N-dimethylformamide, N,N-dimethylacetamide, N-methyl-2-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, \*\*ethylene \*\*glycol\*\*, and diethylene \*\*qlycol\*\* monobutyl \*\*ether\*\*, because these solvents bring a 4395348 classes:1 510/176 1 134/3 1 134/38 1 134/40 1 134/41 1 134/42 1 430/329 1 430/331 1 510/202 1 510/414 1 5 10/505 1 568/763 score: 635 keywords: glycol;ethylene glycol;ethylene glycol;fluoride;acid;glycol;ethylene; ortho, meta and para isomers of xylene and diethylbenzene; trialkyl-substituted aromatic hydrocarbons containing 9 to 20 carbon atoms, such as the 1,2,3-; 1,2,4- and 1,3,5-isomers of trimethyl and triethylbenzene; suitable polar organic solvents include aliphatic ketones containing three to 10 carbon atoms, such as acetone, methylethylketone and methylisobutylketone; monoalkyl \*\*ether\*\*s of \*\*ethylene \*\*glycol\*\* containing three to 10 carbon atoms, such as ethoxyethanol and butoxyethanol; carboxylic acids containing one to four carbon atoms, such as acetic and maleic acid; formamide; N,N-dialkylalkanonylamides containing three to 10 carbon atoms, such as dimethylformamide and dimethylacetamide; N-alkyl lactams containing six to 12 carbon atoms, such as N-methylpyrrolidone; cyclic aliphatic \*\*sulfone\*\*s containing four to 6 carbon atoms, such as \*\*tetramethyl\*\*enesulfone; and the like. The preferred nonpolar, organic solvents are xylene 4765844 classes:1 510/176 1 134/38 1 252/364 1 430/329 1 510/202 1 510/212 1 510/499 1 510/506 score: 628 keywords: glycol;glycol ether;propylene glycol;ethylene glycol;ammonium hydroxide;ammonium hydroxide;hydr oxide; ethylene glycol; propylene glycol; propylene; glycol ether; tetramethyl ammonium; tetramethyl; aqueous; am monium; amine; glycol; ether; ethylene; United States Patent: 4,765,844 FULL TEXT FORMAT --> ( 1of1) United States Patent 4,765,844 , &nbsp et al. August 23, 1988 Solvents for photoresist removal The invention relates to solvent systems which are based on water-soluble amino derivatives and \*\*propylene \*\*glycol\*\* components, for removing photoresists. These solvent systems are comprised of (a) from about 10 to 100% by weight of at least one water-soluble \*\*amine\*\* of the general formula ##STR1##

in which R.sup.1, R.sup.2, R.sup.3, R.sup.4 denote H or alkyl groups

Page 7

(b) from about 0 to 90% by weight of at least one water-soluble \*\*propylene\*\*

n,m denote 0 to 2, and
o denotes 1 to 3; and

```
report 09881552.txt
     **glycol** derivative of the general formula
      in which R.sup.3, R.sup.4 denote H, alkyl or
     ##STR3##
      and P denotes 1 to 3.
  Inventors:
  Merrem; Hans-Joachim(Seeheim-Jugenheim, DE);
Schmitt; Axel(Walluf, DE)
  Assignee:
     the formula
     ##STR7##
      in which R.sup.1, R.sup.2, R.sup.3, R.sup.4 denote H or alkyl groups
n, m denote 0 to 2, and
o denotes 1 to 3;
(b) from about 10 to 90% by weight of at least one water-soluble **propylene**
     **glycol** derivative represented by the formula
     ##STR8##
      in which R.sup.3, R.sup.4 denote H, alkyl or
     groups, and P denotes 1 to 3; wherein said agent is biodegradable, of low toxicity, free of **ammonium **hydroxide** base, relatively non corrosive to
     aluminum and wherein substantially all components of said agent boil at
     temperatures of at least 160.degree. C.
2. An agent as claimed in claim 1, wherein said water-soluble **amine** is
     selected from the group consisting of isopropanolamine and
     N-(2-aminoethyl)-ethanolamine.
3. An agent as claimed in claim 1, wherein said water-soluble **propylene**
     **glycol** derivative is selected from the group consisting of dipropylene
     **glycol** methyl **ether** and tripropylene **glycol** met
     systems, based on
     water-soluble amino derivatives and **propylene **glycol** compounds, that are
     useful for removing photoresists.
Photoresists are used as masks in the production of, via etching or
     implantation processes, integrated component parts, circuit boards, and
     the like. After having served their purposes, the masks are generally removed, frequently by means of agents which, depending on their mode of action, are known as "strippers" or "removers".
Systems of this kind are described, for example, by W. S. de Forest in
     "Photoresist: Materials and Processes" pages 203 et seq., McGraw-Hill Book
     Co., New York, 1975. They contain, e.g., chlorinated hydrocarbons, such as
     methylenechloride; polar solvents, such as dimethylformamide,
     N-methyl-2-pyrrolidone and monoethanolamine; **glycol **ether**s, such as 
**ethylene **glycol** monoethyl **ether**, **ethylene **glycol** monobutyl **ether** and the
     acetates thereof; strong bases, such as **tetramethyl ammonium**
               4617251
classes:1 430/256 1 134/38 1 430/258 1 430/331 1 510/176 1 510/212 1 510/407 1 510/493 1 510/499 1 510/50
0 1 510/501 1 510/505
score: 620
keywords: glycol;nmp;nmp;aqueous;acid;amine;glycol;ether;
     pletion of the stripping operation by the use of
      **aqueous** materials, such as deionized water.
Now it has been discovered, in accordance with the present invention, that
     the foregoing problems can be overcome with a stripping composition that
     includes a select **amine** compound and an organic polar solvent, the
     composition being essentially free of phenol compounds and halogenated
     hydrocarbon compounds. In the practice of the invention, an organic
     polymeric material is removed from a substrate by contacting the organic
     polymeric material with the stripping composition as described above.
     Surprisingly, it has been found that the stripping composition is highly
     effective in removing organic polymeric materials from substrates,
     although as noted it is essentially free of phenol compounds and
     halogenated hydrocarbon compounds, the use of which is taught in the art.
     Furthermore, because the stripping composition is essentially free of s
```

----**-** 5955410

classes:1 510/202 1 134/2 1 134/3 1 134/38 1 134/40 1 134/41 1 510/206 1 510/417 1 510/488 1 510/499 1 51 0/500 1 510/506

score: 606

keywords: pyrrolidone;glycol;glycol ether;propylene glycol;ethylene glycol;nmp;pyrrolidone;ammonium hydro
xide;ammonium hydroxide;hydroxide;ethylene glycol;propylene;glycol ether;pyrrolidone;nmp
;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- the proportions indicated. In the formulations of the present invention, the \*\*pyrrolidone\*\* constituent is believed to achieve an enhanced varnish removing or cleaning ability when combined with a \*\*propylene \*\*glycol\*\* aliphatic \*\*ether\*\* compound, including an acetate thereof. Such combinations of N-methyl-2-pyrrolidone and a \*\*propylene \*\*glycol\*\* solvent are utilized in the various formulations of the present invention.
- The alkanolamine component of the present invention is preferably an ethanolamine and, most preferably, monoethanolamine. Monoethanolamine is an extremely effective solvent for varnish and the type of baked on carbon residue which is typical of various parts of internal combustion engines following substantial and continuous use under normal as well as extreme operating conditions. In the appropriate proportions, monoethanolamine will form an effective soap with one or more aliphatic acid in a single phase cleaning system in
- y be used with various aluminum alloys without damage thereto. Furthermore, the corrosive characteristics of such caustic mixtures has always been a danger to those using such formulations. By use of the present invention, the dangers of corrosive damage as well as other inherent health hazards are avoided.

Generally, the cleaning compositions of the present invention contain no chlorinated solvents. The alkanolamine is used to remove varnish and carbon deposits from various engine parts and will combine with aliphatic fatty acids, when present, to form a soap. \*\*ammonium \*\*hydroxide\*\* may be used in combination with the alkanolamine to further enhance carbon removal. The \*\*propylene glycol \*\*ether\*\*, or an ester thereof, combined with an alkylpyrrolidone, is included as a degreasing substitute for methylene chloride. In the formulations disclosed and described herein, the present invention is generally less volatile and less of a health

----**-** 6323169

classes:1 510/176 1 510/175 1 510/245 1 510/255 1 510/257 1 510/264 1 510/499 score: 602

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;sulfone;amide;glycol;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol;propylene;amide;tetramethyl;alkaline;ammonium fluoride;fluoride;sulfone;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- anediaminetetramethylenephosphonic acid, diethylenetriaminepentamethylenephosphonc acid and \*\*ethylene\*\*diaminetetrakismethylenephosphonic acid are preferable, and particularly preferred is 1,2-propanediaminetetramethylenephosphonic acid.
- The above chelating agents may be used alone or in combination of two or more.
- The concentration of the chelating agent preferably 0.01 to 5% by weight, more preferably 0.05 to 3% by weight based on the total weight of the stripping composition.
- The water-soluble fluorine compounds (c) usable in the present invention may include organic \*\*amine fluoride\*\*s such as \*\*ammonium \*\*fluoride\*\*, acid \*\*ammonium \*\*fluoride\*\* and monoethanolamine \*\*fluoride\*\*, and \*\*tetramethyl ammonium\*\* \*\*fluoride\*\*. The concentration of the water-soluble fluorine compound is preferably 0.001 to 10% by weight, more preferably 0.005 to 5% by weight based on the total weight of the stripping composition.

The organic solvents (d) usable in the present inv

ention may include \*\*ether\*\*
solvents such as \*\*ethylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*ethylene \*\*glycol\*\*
monobutyl \*\*ether\*\*, diethylene \*\*glycol\*\* monomethyl \*\*ether\*\*, diethylene \*\*glycol\*\*
monoethyl \*\*ether\*\*, diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\*
monomethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\* monoethyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\*
monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monomethyl \*\*ether\*\*, dipropylene \*\*glycol\*\*
monoethyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*, diethylene \*\*glycol\*\*
dimethyl \*\*ether\*\* and dipropylene \*\*glycol\*\* dimethyl \*\*ether\*\*; \*\*amide\*\* solvents such
as formamide, monomethylformamide, dimethylformamide, monoethylformamide,

diethylformamide, acetamide, monoethylacetamide, dimethylacetamide, monoethylacetamide, diethylacetamide, N-methylpyrrolidone and N-ethylpyrrolidone; and sulfur compound solvents such as dimethyl \*\*sulfoxide\*\*, dimethyl \*\*sulfone\*\*, diethyl \*\*sulfone\*\*, bis(2-hydroxy) \*\*sulfone\*\* and \*\*tetramethyl\*\*ene \*\*sulfone\*\*. Preferred are dimethyl \*\*sulfoxide\*\*, N,N-dimethy

----- 5563119

classes:1 510/176 1 134/2 1 134/38 1 134/40 1 430/329 1 510/201 1 510/206 1 510/212 1 510/401 1 510/402 score: 553

keywords: alkaline; ammonium hydroxide; ammonium hydroxide; hydroxide; tetraethyl; tetramethyl ammonium; tetram ethyl; alkaline; aqueous; acid; ammonium; amine;

or
 different than R.sub.3. Preferred inhibitors include catechol, pyrogallol,
 anthranilic acid, gallic acid, gallic esters, and the like.
The alkanolamines which are useful in the compositions of the invention are
 the lower alkanol \*\*amine\*\*s which include monoethanolamine, diethanolamine,
 mono-, di- and tri-isopropanolamine, and the like. Preferred is
 monoethanolamine alone or in combination with other alkanolamines.
The tetraalkylammonium \*\*hydroxide\*\* compounds which are useful in the
 invention are \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), \*\*tetraethyl ammonium\*\*
 \*\*hydroxide\*\*, diethyl dimethyl \*\*ammonium \*\*hydroxide\*\*, and the like.
 Advantageously, the hydrates of the \*\*hydroxide\*\* compounds are utilized. The
 most preferred \*\*hydroxide\*\* compound is TMAH.5H.sub.2 O.
The preferred stripping composition consists essentially of about 73% by
 weight of monoethanolamine, about 18% by weight of water, about 5% by
 weight of inhibitor and about 4% by weight of TMAH.5H.s

----- 5962383 classes:1 510/164 1 134/42

score: 553

keywords: ph;pyrrolidone;glycol;glycol ether;propylene glycol;ethylene glycol;pyrrolidone;ammonium hydroxide;weak;ammonium hydroxide;hydroxide;ethylene glycol;propylene glycol;propylene;glycol ether;pyrrolidone;tetramethyl ammonium;tetramethyl;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

such as \*\*weak acid\*\*s and their conjugate bases, for example, acetic acid and \*\*ammonium\*\* acetate. Preferred for use as such components are acetic acid, boric acid, citric acid potassium biphthalate, mixtures of \*\*ammonium\*\* chloride and \*\*ammonium\*\* acetate, especially a 1:1 mixture of these two salts, and mixtures of acetic acid and ammonia and other \*\*amine\*\*s.
The following examples are illustrative of the present invention and are not meant to, and should not be taken to, limit the scope of the invention.

EXAMPLE 1
An optical mold is selected that has been contaminated with a diethylene

\*\*glycol\*\* bisallyl carbonate (DEGBAC) based monomer. The polymer is hardened
on the external side of the mold and the mold is further contaminated with
fingerprint oils and dirt. The contaminated mold is immersed in a solution
of 2.5% \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\*, 15% potassium \*\*hydroxide\*\*, 15% sodium
\*\*hydroxide\*\* and 67.5% water at 150 to 160.degree. F. (ca 65.d

----- 5202049 classes:1 510/206 1 134/38 1 134/39 1 134/40 1 510/405 1 510/421 1 510/424 1 510/437 1 510/499 1 510/506 score: 540

keywords: ph;glycol;ethylene glycol;ethylene glycol;acid;glycol;ether;ethylene;

```
- es Cited [Referenced By]
U.S. Patent Documents
4081395Mar., 1978Talley252/534.
4554026Nov., 1985Cosper et al.134/38.
4565644Jan., 1986Smith et al.252/153.
4678605Jul., 1987Geke et al.252/547.
4814108Mar., 1989Geke et al.252/545.
```

```
4857114Aug., 1989Brumbaugh et al.252/DIG.
4915864Apr., 1990Kita et al.252/132.
5096610Mar., 1992Bingham252/162.
  Primary Examiner: Lieberman; Paul
  Assistant Examiner: Parks; William S.
Parent Case Text
This is a continuation of copending application Ser. No. 07/610,196 filed
     on Nov. 6, 1990 now abandoned.
Claims
What is claimed is:
1. A sealer finish remover composition comprising effective amounts of (a)
     a solvent selected from diethylamine, diethanolamine, monoethanolamine,
     **ethylene **glycol** monobutyl **ether**, **ethylene **glycol** phenyl **ether**, and
     mixtures thereof, (b) water, (c) about 4-8 weight % of a metasilicate, and (d) about 1-30 weight % of a linear, 6-10 carbon, organic compound
     selected from caprylic acid, n-o
              4921626
classes:1 252/794 1 216/90 1 216/99 1 252/793
score: 528
keywords: ph;glycol;propylene glycol;weak;propylene glycol;propylene;fluoride;buffered;acid;ammonium;glyc
     63 -
4585514Apr., 1986Scallan156/663.
  Primary Examiner: Powell; William A.
  Attorney, Agent or Firm: Lamb; Charles G.
Claims
What is claimed:
1. An etching composition for etching glass comprising:
from about 16% to about 30% by weight of **ammonium** bifluoride;
from about 2% to about 4% by weight of xanthum gum;
from about 45% to about 60% by weight of water; and,
from about 20% to about 24% by weight of **propylene **glycol**.
2. The etching composition of claim 1, further comprising less than 2% by
     weight of phosphoric acid.
3. The etching composition of claim 2, having a pH of about 5.0.
4. A process for making a glass etching composition comprising the steps
    of:
adding **ammonium** bifluoride to water in a first vessel while agitating the
     water;
continuing to agitate the **ammonium** bifluoride-water mixture until all of
     the **ammonium** bifluoride is completely dissolved in the water;
adding xanthum gum to **propylene **glycol** while agitating the **propylene **glycol**
     in a se
              4250193
classes:1 514/772 1 424/701 1 424/73 1 514/777
score: 522
keywords: glycol;glycol ether;propylene glycol;ethylene glycol;hydroxide;ethylene glycol;propylene glycol
;propylene;glycol ether;aqueous;acid;amine;glycol;ether;ethylene;
     1 compounds
     (A).about.(H) underwent no substantial hydrolysis. To the contrary, both
     (I) and (J) had at least more than 90% hydrolyzed. In this connection, it
     has been found that the hydrolysis of each compound other than the
     compounds (A).about.(H) is in a similar order
Some preparation examples of the novel compounds of the present invention
     will now be given.
Preparation Example 1
0.5 grams of a boron trifluoride **ether** complex were added to 31 grams of
     **ethylene **glycol** and were fully mixed under agitation therewith. To the
     resultant mixture were gradually added dropwise 102 grams of ethylglycidyl
     **ether** at room temperature over a period of 9.5 hours. The resultant
     product was then heated at about 100.degree. C. for 3 hours and was added
     with 10 grams of anhydrous sodium carbonate, followed by sufficient
     stirring and mixing. Thereafter, the undissolved matters were filtered
     off. The formed oily liquid was rectified under reduc
```

report 09881552.txt 114 grams of 2-propenylqlycidyl \*\*ether\*\* at room temperature over a period of 10 hours. The resultant product was then heated at about 100.degree. C. for 3 hours and was added with 50 cc of potassium carbonate solution, followed by sufficient stirring and mixing. Thereafter, the formed product from which the \*\*aqueous\*\* phase had been removed was rectified under reduced pressures to obtain 68 grams of bis-(2-propenylglyceryl)-tetramethylene \*\*ether\*\*. Preparation Example 4 0.5 grams of sodium \*\*hydroxide\*\* were added to 51 grams of diethylene \*\*glycol\*\* and were fully mixed under agitation therewith. To the resultant mixture were gradually added dropwise 102 grams of ethylglycidyl \*\*ether\*\* at room temperature over a period of 9.5 hours. The resultant product was then heated at about 100.degree. C. for 3 hours and was added with 10 grams of anhydrous sodium carbobate, followed by sufficient stirring and mixing. Thereafter the undissolved matters wer ith. To the resultant mixture were gradually added dropwise 130 grams of butylglycidyl \*\*ether\*\* at 100.degree. C. over a period of 9.5 hours. The resultant product was at room temperature and was added with 100.degree. cc of sodium carbonate solution, followed by sufficient stirring and mixing. Thereafter, the \*\*aqueous\*\* phase had been removed. The formed oily liquid was rectified under reduced pressures to obtain 102 grams of bis-(butylglyceryl)-triethylene \*\*glvcol \*\*ether\*\*. Preparation Example 7 0.5 grams of sodium \*\*hydroxide\*\* were added to 82 grams of \*\*tetraethyl\*\*ene \*\*glycol\*\* and were sufficiently mixed under agitation therewith. To the resultant mixture were gradually added dropwise 128 grams of amylglycidyl \*\*ether\*\* at 100.degree. C. over a period of 10 hours. The resultant product was at room temperature and was added with 50 cc of water, followed by sufficient stirring and mixing. Thereafter, the formed product from which the \*\*aqueous\*\* Bis-(propylglyceryl)-triethylene \*\*ether\*\* Polyoxyethylene-cured castor oil derivative 0.4 0.1 Perfume Component B 20 Refined water 0.25 Succinic acid 0.05 Sodium succinate Component C 54.2 Refined water

To the components A dissolved at 60.degree.-70.degree. C. were gradually adde the components B at room temperature. The refined water C was then added to the resultant solution, followed by filtration at room temperature. Thus the product was formed.

EXAMPLE 2--COSMETIC REMOVER

Component A
Ethanol 15
\*\*propylene \*\*glycol\*\* 5
Bis-(propylglyceryl)-tetraethylene \*\*ether\*\*
10
Polyoxyethylene-cured castor oil derivative

----- 6191086 classes:1 510/175 1 134/12 1 134/13 1 134/3 1 134/2214 1 510/176 1 510/178 1 510/201 1 510/202 1 510/203 1 510/204 1 510/210 1 510/212 1 510/225 1 510/245 1 510/254 1 510/256 1 510/257 1 510/259 1 510/412 1 510/477 1 510/480 1 510/504

score: 518

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;tetramethyl ammonium fluoride;glycol;ethylene glycol;nmp;ammonium hydroxide;weak;ammonium hydroxide;hydroxide;ethylene glycol;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;ammonium fluoride;fluoride;nmp;aqueous;acid;ammonium;glycol;ethylene;

reof. Preferred acid \*\*fluoride\*\*s include hydrogen \*\*fluoride\*\*,
(HF), and perfluoric acid. Preferred \*\*fluoride\*\* salts are \*\*ammonium \*\*fluoride\*\*,
\*\*ammonium\*\* bifluoride, \*\*ammonium\*\* difluoride and tetraalkyl \*\*ammonium \*\*fluoride\*\*
salts such as \*\*tetramethyl \*\*ammonium \*\*fluoride\*\* and \*\*tetraethyl ammonium\*\*
\*\*fluoride\*\*. Preferably, the fluorine-containing compound will comprise about

- 0.5 to 10 wt. % based upon the weight of the composition.

  The polyammonium salts may be prepared by reacting a polyamine with HF to form the polyammonium \*\*fluoride\*\* salt. For example, HF may be reacted with an \*\*aqueous\*\* solution of \*\*ethylene\*\* diamine to form an \*\*ethylene\*\*diammonium difluoride. Alternatively, an excess amount of HF may be reacted with the polyamine to form the polyammonium salt. Preferred polyammonium salts include \*\*ethylene\*\*diammonium difluoride and diethylenetriammonium trifluoride.
- It is believed that the fluorine-containing compound aids in removal of contaminates by slight
- lamine
  - salt has been found by the present applicants to be particularly useful as a cleaning composition.
- The pH of the cleaning composition preferably ranges from weakly acidic to slightly basic so as to not attack or corrode the metal used in the microcircuit fabrication. If the cleaning composition is either too caustic or too acidic, the metal layers on the substrate are subject to attack. Therefore, the components in the cleaning composition should be admixed in appropriate concentrations to provide a composition having a pH with a preferred range from about 2 to 9, and more preferably from about 2 to 6.
- In a preferred embodiment of this invention, the fluorine-containing compound is admixed with a composition comprising water, a hydroxylamine salt and at least one basic compound selected from the group consisting of: \*\*amine\*\*s, quaternary \*\*ammonium \*\*hydroxide\*\*s and \*\*ammonium \*\*hydroxide\*\*. Such compositions are described in co

4822723

classes:1 430/331 1 430/302 1 430/309 1 510/171 1 510/176 1 510/206

score: 513

keywords: glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;aqueous;acid
;amine;glycol;ether;ethylene;

- nufacture of lithographic printing plates such as, Alcoa 3003 and Alcoa 1100, which may or may not have been pretreated by standard graining and/or etching and/or anodizing techniques as are well known in the art, and also may or may not have been treated with a composition, such as polyvinyl phosphonic acid, suitable for use as a hydrophilizing layer for lithographic plates is coated with a light sensitive polymeric diazonium salt or photopolymer containing composition. Such compositions may also contain binding resins, such as polyvinyl formal resins, colorants, acid stabilizers, surfactants, exposure indicators or other art recognized ingredients. The photosensitive sheet material is then exposed to a suitable radiation source through a mask or transparency, and the exposed sheet then developed for removal of the non-image photosensitive materials. The photosensitive coating mixture is usually prepared in a solvent
- hem, to remove the non-image areas, using a developer which contains the aforementioned ingredients in \*\*aqueous\*\* mixture.
- The developr contains a \*\*propylene \*\*glycol\*\* monomethyl \*\*ether\*\* or dipropylene 
  \*\*glycol\*\* monomethyl \*\*ether\*\* component. In the preferred embodiment this 
  component is present in an amount of from about 25.0% to about 75.0% by 
  weight of the developer. A more preferred range is from about 35.0% to 
  about 65.0% and most preferably from about 45.0% to about 55.0%.
- The developer also contains from about 0.05% to about 5.0% by weight of the developer of \*\*ethylene\*\* carbonate or \*\*propylene\*\* carbonate. A more preferred range is from about 0.1% to about 1.0% and most preferably from about 0.3% to about 0.7%.
- The developer further contains from about 1.0% to about 30.0% by weight of the developer of \*\*ethylene \*\*glycol\*\* diacetate. A more preferred range is from about 5.0% to about 20.0% and most preferably from about 10.0% to about 15.0%.

The

- based on the weight of the developer. In one preferred embodiment this component is a mixture of glycerin and \*\*ethylene \*\*glycol\*\* in a relative ratio of about 1:15 to about 200:1.
- The developer contains 0.05% to about 5.0% based on the weight of the developer of benzoic or citric acid. A more preferred range is from about 0.1% to about 1.0% and most preferably from about 0.4% to about 0.6%.

  Page 13

The developer contains from about 0.5% to about 10.0% based on the weight of the developer of benzyl alcohol or phenoxyethanol. A more preferred range is from about 1.0% to about 5.0% and most preferably from about 2.0% to about 4.0%. In the preferred embodiment, the developer of the present invention preferably contains optional minor amounts of a conventional antifoam component which aids in processing of the printing plate. This component, when it is used is preferably present in an amount of from about 0.005% to about 0.075% by weight of the de t is observed in these

developed plates.

As a comparison, Newplates S plates developed with Graphex Lacquer (available from Graphex litho System of Milwaukee, WI 53214) using the same processor parameters is used as a control. The lithographic properties are given below:

This Comparative Lithographic Property Developer Lacquer Solid/ghost on a 21 step Stouffer 5/9 Resolution: UGRA Circle (microns) 8-12 12 - 15UGRA 150 lines per inch 1-97%

2-98%

Impressions to Roll-up

15 .+-. 5 30 .+-. 5

Length of Run (abrasive conditions) 220M 75M Redeposit None Yes Odor Strong Weak

The experim

4822514

classes:1 510/400 1 510/406 1 510/421 1 510/422 1 510/423 1 510/433 1 510/481

keywords: alkaline; amide; glycol; amide; alkaline; aqueous; acid; amine; glycol; ether; ethylene;

61790-44-1 about 0.8% tall oil Tripropylene \*\*glycol\*\* 112-35-6 Dowanol about 3.5% monomethyl \*\*ether\*\* TPM Triethanolamine 102-71-6 about 1.5% Coconut diethanolamide 68603-42-9 Clindrol about 100 C-G 0.75% Polyalkoxylated 52624-57-4 Pluracol V-10 about 1.0% trimethylolpropane Disodium EDTA 64-02-8 about 0.08% Fragrance about 0.36% P-250 about 0.4% about 0.04% Geraniol 500 Water Q.S.

While the cleaning and selective gloss or shine imparting or restoring mechanism of the novel composition of the invention is not completely

4248724 classes:1 508/208 1 252/387 1 252/396 score: 504

keywords: glycol;glycol ether;ethylene glycol;ethylene glycol;propylene;glycol ether;acid;glycol;ether;et
hylene;

```
11,396,52 A,52 R
References Cited [Referenced By]
U.S. Patent Documents
3532624Oct., 1970Cekada, Jr.252/49.
3770633Nov., 1973Holley252/49.
4088591May., 1978Brown, Jr. et al.252/49.
  Primary Examiner: Howard; Jacqueline V.
  Attorney, Agent or Firm: Krass & Young
Claims
The embodiments of the invention in which an exclusive property or
     privilege is claimed are defined as follows:
1. A penetrating and lubricating composition consisting essentially of:
(a) from 0.7% to 1.6% by volume of a dimethyl siloxane polymer; and
(b) a **glycol **ether** selected from the group consisting of **ether**s of **ethylene**
    **glycol**, **ether**s of **propylene **glycol**, **ether**s of diethylene **glycol**, and
    **ether**s of dipropylene **glycol**.
2. The composition of claim 1 wherein said **glycol **ether** is **ethylene **glycol**
     butvl **ether**.
3. The composition of claim 1 wherein said dimethyl siloxane polymer has an
     average viscosity at 25.degree. centigrade of about 100 centistokes.
4. A penetrating and lubricatin
     ition is applied to the exterior of the corroded parts
     that are to be separated. The extremely low surface tension of the **glycol**
      **ether** solvent--on the order of from 25 to 30 dynes per centimeter at
     25.degree. centigrade--allows the formula to seek out and seep into the
     most minute cracks between the surfaces to be freed and dissolve any rust
     or corrosion present. The dimethyl siloxane polymer is carried into the
     cracks with the **glycol **ether** in which it is dissolved and remains on the
     surfaces as a lubricating and corrosion preventing coating after the
     **glycol **ether** has evaporated.
For the purposes of this invention, **glycol **ether** will be defined as the **ether**s of **ethylene**, **propylene**, diethylene, or dipropylene **glycol**. These
     are produced by the reaction of alkylene oxides with alcohols or phenols,
     the **glycol **ether**s used in the following examples were obtained from the
     Dow Chemical Company which markets them under the trade name Do
      fluid ounce of Dow Corning 200 fluid of 100 centistokes viscosity (at
     25.degree. centigrade) was mixed with one gallon of Dowanol EB (ethylene **glycol** butyl **ether**). This formulation was found to have superior
     penetrating lubricating properties when applied to corroded metallic
     parts.
EXAMPLE 2
A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes
     viscosity and one gallon of Dowanol DE (diethylene **glycol** ethyl **ether**) was
     made and tested with good results.
A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes
     viscosity and one gallon of Dowanol PM (propylene **glycol** methyl **ether**) was
     made and tested with good results.
EXAMPLE 4
A mixture of one fluid ounce of Dow Corning 200 fluid of 100 centistokes
     viscosity and one gallon of Dowanol DPM (dipropylene **glycol** methyl **ether**)
was made and tested with good results.

The above examples are listed in order of declining overall efficiency as
     pene
                5571447
classes:1 510/206 1 252/793 1 430/331 1 510/188
score: 503
```

keywords: ammonium fluoride;alkaline;ph;ammonium fluoride;glycol;propylene glycol;ethylene glycol;hydroxi de;ethylene glycol;propylene glycol;propylene;alkaline;ammonium fluoride;fluoride;buffered;fluoroboric;aq ueous;acid;ammonium;glycol;ethylene;

- /or dimethylsulfoxide. Advantageously, the compositions of the invention are free of \*\*ethylene\*\* Page 15

report09881552.txt \*\*glycol\*\* and hydrofluoric acid. The compositions of the invention advantageously contain a corrosion inhibitor. It is a general object of the invention to provide a stripping composition having a controlled etch and sidewall polymer removal capability. It is another object of the invention to provide a stripping composition which is free of \*\*ethylene \*\*qlycol\*\* and hydrofluoric acid. It is yet another object of the invention to provide a stripping composition which is water soluble having high resistance to metal corrosion. It is still another objection of the invention to provide a method for stripping which provides for a controlled oxide etch. BRIEF DESCRIPTION OF THE DRAWINGS FIG. 1 illustrates that the overall selective etching of the compositions of the invention with respect to \*\*tetraethyl\*\*orthosilcate (TEOS) vs. silicon is superior over a wide range oxide surfaces. The composition are also effective in removing many forms of resist ash and etch residues without the presence of hydrogen \*\*fluoride\*\*. The stripping and cleaning compositions of the invention contain about 60 to 85% by weight of composition of a polyhydric alcohol which excludes \*\*ethylene \*\*glycol\*\*, for example \*\*propylene \*\*glycol\*\*, glycerol, polyethylene \*\*glycol\*\*, etc. Fluoboric acid in an amount of about 0.5 to 10% by weight of composition is an essential component of the composition. Fluoboric acid maintains a stable \*\*fluoride\*\* ion source with \*\*ammonium \*\*fluoride\*\* or other \*\*fluoride\*\* salts and provides the needed low pH of the compositions. Polar solvents which can be used in the invention include water, \*\*amide\*\*s, aliphatic esters, lactates, cyclic \*\*lactone\*\*s, and the like. The use of about 5 to 40% by weight of a polar solvent, especially water and/or dimethyl 4622303 classes:1 435/243 1 435/3 1 435/246 1 435/2557 1 435/812 1 514/937 1 514/943 1 514/945 1 516/134 keywords: glycol;glycol ether;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene ;glycol ether;aqueous;acid;glycol;ether;ethylene; n accordance with claim 8 wherein the polyglycol \*\*ether\*\*s contain an average of from 1 to 3 \*\*ethylene glycol \*\*ether\*\* groups. 10. A fermentation broth in accordance with claim 7 wherein from about 0.005 to about 1 g/l of said polyglycol \*\*ether\*\*s are present in the fermentation broth. 11. A fermentation broth in accordance with claim 10 wherein from about 0.01 to about 0.2 g/l of said polyglycol \*\*ether\*\*s are present in the fermentation broth. 12. A fermentation broth in accordance with claim 7 wherein the alcohol component of the polyglycol \*\*ether\*\*s is a mixture of oleyl alcohol and cetyl alcohol components in a ratio by weight of from about 3:1 to about 1:1, and wherein the polyglycol \*\*ether\*\*s contain an average of 2 \*\*ethylene \*\*glycol\*\* \*\*ether\*\* groups. Description BACKGROUND OF THE INVENTION 1. Field of the Invention The present invention relates to compounds for the suppression of foaming in yeast fermentation without the inhibition of yeast growth or t myristyl and stearyl alcohol can also be present in admixture with oleyl alcohol and cetyl alcohol, their quantity should not exceed 10% by weight in either case. Polyglycol \*\*ether\*\*s in which from about 30 to about 80% of the alcohol component consists of oley1 alcohol are particularly suitable. More highly unsaturated alcohols are unsuitable and should preferably be absent.

The number of polyglycol \*\*ether\*\* groups amounts on average to from 1 to 4 and preferably to from 1 to 3. As normal with commercial alkoxylation products, this figure represents a statistical average, i.e. the alkoxylation products may also contain small amounts of alcohols alkoxylated to a higher or lower degree, including unsubstituted alcohols, corresponding to a statistical distribution. The \*\*glycol \*\*ether\*\* groups can consist of \*\*ethylene glycol \*\*ether\*\* groups (EO) or \*\*propylene glycol \*\*ether\*\*

groups (PO) or both. They preferably consist of EO-groups; their number

5419779 classes:1 134/38 1 134/2 1 134/40 1 252/364 1 252/387 1 252/392 1 252/396 1 510/176 1 510/202 1 510/206 1 510/212 1 510/499 score: 496 keywords: hydroxide; aqueous; acid; amine; e like. Specific examples of organic polymeric materials include positive resists containing phenolformaldehyde resins or poly (p-vinylphenol), polymethylmethacrylate-containing resists, and the like. Examples of plasma processing residues (sidewall polymer) include among others; metal-organic complexes and/or inorganic salts, oxides, \*\*hydroxide\*\*s or complexes which form films or residues either alone or in combination with the organic polymer resins of a photoresist. The organic materials and/or SWP can be removed from conventional substrates known to those skilled in the art, such as silicon, silicon dioxide, aluminum, aluminum alloys, copper, copper alloys, etc. The hydroxylamine utilized in the invention is commercially available as a 50% \*\*aqueous\*\* solution from Nissin Chemical Industry Company, Tokyo, Japan. The effectiveness and unexpected nature of the stripping compositions of the invention is illustrated, but not lim 6465403 classes:1 510/175 1 510/176 1 510/178 1 134/3 1 438/745 score: 486 keywords: alkaline;ph;sulfoxide;glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;tetraethyl;tetrame thyl ammonium; tetramethyl; alkaline; sulfoxide; aqueous; acid; ammonium; amine; glycol; ether; ent \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.1 weight percent trans-(1,2-cyclohexylenedinitrilo) tetraacetic acid (CyDTA), 0.07 weight percent of the non-ionic surfactant Surfynol-465, 0.14 weight percent (calculated as % SiO.sub.2) \*\*tetramethyl\*\*ammonium silicate (TMAS) and 3 weight percent glycerol added with the remainder of this solution being water and has a pH of about 12.1. \*\*aqueous\*\* solution "M1" was prepared with 1.2 weight percent \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.45 weight percent trans-(1,2-cyclohexylenedinitrilo)-tetraacetic acid (CyDTA), 0.14 weight percent (calculated as % SiO.sub.2) \*\*tetramethyl\*\*ammonium silicate (TMAS), 18.5 weight percent hydroxylamine and 0.07 weight percent of the non-ionic surfactant Surfynol-465 (remainder of this solution being water) and has a pH of about 12.1. \*\*aqueous\*\* solution "P1" was prepared with 2.2 weight percent \*\*tetramethyl\*\*ammonium \*\*hydroxide\*\* (TMAH), 0.11 weight percent tra 5381807 classes:1 134/2 1 134/38 1 134/40 1 134/42 1 430/258 score: 480 keywords: amide; glycol; propylene glycol; ethylene glycol; ethylene glycol; propylene glycol; propylene; amide; acid; glycol; ethylene; atoms. Preferred alkanolamines suitable for use in the present invention can be represented by the chemical formula R.sub.1 R.sub.2 --N--CH.sub.2 CH.sub.2 --O--R.sub.3 wherein R.sub.1 and R.sub.2 can be H, CH.sub.3, CH.sub.3 CH.sub.2 or CH.sub.2 CH.sub.2 OH and R.sub.3 is CH.sub.2 CH.sub.2 OH. Examples of suitable alkanolamines include monoethanolamine,

diethanolamine, triethanolamine, tertiarybutyldiethanolamine

2-amino-(2-ethoxyethanol), and 2-amino(2-ethoxy)propanol.

isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine,

Polar solvents suitable for use in the present invention include \*\*ethylene\*\*

N-substituted \*\*pyrrolidone\*\*, \*\*ethylene\*\*diamine, and \*\*ethylene\*\*triamine. Additional polar solvents as known in the a

----- 5498293 classes:1 134/3 1 134/2 score: 480

keywords: alkaline;ph;glycol;glycol ether;propylene glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;propylene glycol;propylene;glycol ether;tetraethyl;tetramethyl ammonium;tetramethyl;alkaline;fluoride;a queous;acid;ammonium;amine;glycol;ether;

on of a \*\*propylene glycol \*\*ether\*\* organic solvent,
particularly a \*\*propylene \*\*glycol\*\* monoalkyl \*\*ether\*\* such as n-butoxypropanol,
is also beneficial to enhance detergency and increase the metal holding
capacity of the cleaner composition. A preferred formulation will contain
both a \*\*propylene \*\*glycol\*\* monoalkyl \*\*ether\*\* organic solvent and a metal
chelating agent, preferably \*\*ethylene\*\*diaminetetraacetic acid and
n-butoxypropanol.

In the cleaner compositions used in the process of this invention, the

\*\*alkaline\*\* component will generally be present in an amount of up to about
25% by weight of the composition, generally in an amount of from about
0.05 to about 10% by weight, and preferably in an amount of from about 0.1
to about 5% by weight. The amphoteric surfactant will generally be present
in an amount of up to about 10% by weight, generally in an amount of from
about 0.001 to about 10% by weight, and preferably in an amount of from
about

----- 4836950

classes:1 510/170 1 510/172 1 510/407 1 510/412 1 510/413 1 510/505 1 510/506

score: 477

keywords: ph;glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene;glycol;et her;ethylene;

		0	no		
methyl **ethe	r**				
Ethanol	15.80	8.80	19.4	0	
				no	no
Ethyl acetate	15.80	5.30	7.20	yes	
**ethylene**				<b>1</b>	
	19.00		4.10	yes	
**ethylene **				1.00	
00	15.90			n	
	13.50	4.70	10.0	yes	
ethyl **ether	** 200+	2+0		yes	
	14.90		0 00	no	
					no
Isophorone	16.90	8.20	7.40	no.su	
				_	yes
Methanol	15.10	12.30	22.3	0	
				no	no
Methylene chl	oride				
	18.20	6.30	6.10	yes	
Nmethyl-2-p	yrro-				
	18.00	12.30	7.20	yes	
lidone				-	
Nitrobenzene	20.00	8.60	4.10	yes	
2-nitropropan	e			•	
	16.20	12.10	4.10	ves	
**propylene**				2	
FF/	20.00		4.10	no	yes
**propylene *					100
propyrene	15.60				
	15.00	0.5			

---- 6326348

 ${\tt classes:1\ 510/428\ 1\ 510/119\ 1\ 510/122\ 1\ 510/125\ 1\ 510/235\ 1\ 510/311\ 1\ 510/357\ 1\ 510/424\ 1\ 510/426\ 1\ 51$ 

score: 449

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd Page 18

roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;a
cid;ammonium;amine;glycol;ether;ethylene;

- queous liquid phase is one selected from the alkali metal salts of alkylbenzene sulfonic acids in which the alkyl group contains from about 10 to 16 carbon atoms, in straight chain or branched chain configuration. (See U.S. Pat. Nos. 2,220,099 and 2,477,383, incorporated herein by reference.) Especially preferred are the sodium and potassium linear straight chain alkylbenzene sulfonates (LAS) in which the average number of carbon atoms in the alkyl group is from about 11 to 14. Sodum C.sub.11 -C.sub.14 LAS is especially preferred.
- The alkylbenzene sulfonate anionic surfactant will be dissolved in the nonaqueous liquid diluent which makes up the second essential component of the nonaqueous phase. To form the structured liquid phase required for suitable phase stability and acceptable rheology, the alkylbenzene sulfonate anionic surfactant is generally present to the extent of from about 30% to 65% by weight of the liquid phase.
- erein, i.e., the peroxygen bleaching agents, sodium perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.
- A preferred type of nonaqueous, low-polarity solvent for use in the compositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred.

Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.s

---- 5880062

classes:1 503/201 1 347/56 1 347/98 1 347/105 1 427/152 1 503/205 1 503/206

score: 449

keywords: ph;glycol;propylene glycol;ethylene glycol;ethylene glycol;propylene glycol;propylene;tetraethyl;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

- xture of water and other ink components, the concentration of desensitizing agent may be limited to the amount soluble in the ink. Typical concentrations of desensitizing agent in the ink are at least about 0.5 percent by weight, typically from about 1 to about 40 percent by weight, preferably from about 2 to about 20 percent by weight, more preferably from about 2 to about 10 percent by weight, although the amount can be outside these ranges.

The liquid vehicle of the inks of the present invention may consist of water, or it may comprise a mixture of water and a miscible organic component, such as \*\*ethylene \*\*glycol\*\*, \*\*propylene \*\*glycol\*\*, diethylene \*\*glycol\*\*, glycerine, dipropylene \*\*glycol\*\*, polyethylene \*\*glycol\*\*s, polypropylene \*\*glycol\*\*s, \*\*amide\*\*s, such as urea and substituted ureas, \*\*ether\*\*s, carboxylic acids, esters, alcohols, organosulfides, organosulfoxides, \*\*sulfone\*\*s, alcohol derivatives, carbitol, butyl carbitol, cellusolve, \*\*ether\*\* de

4239661

classes:1 438/471 1 134/2 1 134/38 1 134/42 1 430/326 1 430/331 1 438/329 1 438/745 1 438/754 1 510/175 1 510/372 1 510/373 1 510/421 1 510/434 1 510/504 score: 447

keywords: alkaline;ph;glycol;ammonium hydroxide;ammonium hydroxide;hydroxide;alkaline;aqueous;acid;ammonium;glycol;ether;

material of a positive-working photoresist in an organic solvent. The photoresist film was heated about 20 minutes at 80.degree. C. to evaporate the organic solvent and to suppress fogging. Later, a light was projected through a prescribed photomask aligned with the photoresist. Those portions of the photoresist on which a light was projected were etched by a 2.44% \*\*aqueous\*\* choline solution while those portions of the photoresist

on which a light was not projected were left, thereby causing the surface of the silicon oxide film to be selectively exposed. An image pattern produced had a distinctly defined outline and a high precision.

The above-mentioned intermediate product was again heated 20 minutes at 120.degree. C. to attain a closer attachment between the silicon oxide film and the remaining portion of the photoresist film. Later, the exposed silicon oxide film was etched out with dilute hydrofluoric acid. Those portion

----- 6015781

classes:1 510/302 1 510/276 1 510/367 1 510/421 1 510/422 1 510/506

score: 442

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd
roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;a
cid;ammonium;amine;glycol;ether;ethylene;

- li metal salts of alkylbenzene sulfonic acids in which the alkyl group contains from about 10 to 16 carbon atoms, in straight chain or branched chain configuration. (See U.S. Pat. Nos. 2,220,099 and 2,477,383, incorporated herein by reference). Especially preferred are the sodium and potassium linear straight chain alkylbenzene sulfonates (LAS) in which the average number of carbon atoms in the alkyl group is from about 11 to 14. Sodum C.sub.11 -C.sub.14 LAS is especially preferred.

The alkylbenzene sulfonate anionic surfactant will be dissolved in the nonaqueous liquid diluent which makes up the second essential component of the nonaqueous phase. To form the structured liquid phase required for suitable phase stability and acceptable rheology, the alkylbenzene sulfonate anionic surfactant is generally present to the extent of from about 30% to 65% by weight of the liquid phase. More preferably, the alkylbenzene sulfonate

perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.

A preferred type of nonaqueous, low-polarity solvent for use in the compositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred.

Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\* mono C.sub.2 -C.sub.6 a

----- 6008181

classes:1 510/426 1 510/424 1 510/428 1 510/506 1 560/76 1 568/458 1 568/882

score: 440

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

 erial be capable of actually dissolving all of the detergent composition components added thereto.

The nonaqueous organic materials which are employed as solvents herein are those which are liquids of low polarity. For purposes of this invention, "low-polarity" liquids are those which have little, if any, tendency to dissolve one of the preferred types of particulate material used in the compositions herein, i.e., the peroxygen bleaching agents, sodium perborate or sodium percarbonate. Thus relatively polar solvents such as ethanol should not be utilized. Suitable types of low-polarity solvents useful in the nonaqueous liquid detergent compositions herein do include non-vicinal C.sub.4 -C.sub.8 alkylene \*\*glycol\*\*s, alkylene \*\*glycol\*\* mono lower alkyl \*\*ether\*\*s, lower molecular weight polyethylene \*\*glycol\*\*s, lower molecular weight methyl esters and \*\*amide\*\*s, and the like.

A preferred type of nonaqueous, low-polarity solvent for use in the

ompositions herein comprises the non-vicinal C.sub.4 -C.sub.8 branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred. Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\* mono C.sub.2 -C.sub.6 alkyl \*\*ether\*\*s. The specific examples of such compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*, and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve. Another preferred type of nonaqueous, low-polarity organic solvent usefu 6060443 classes:1 510/426 1 510/424 1 510/428 1 560/76 1 568/458 1 568/882 score: 436 keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd roxide; ethylene glycol; propylene glycol; propylene; pyrrolidone; amide; alkaline; buffered; sulfoxide; aqueous; a cid; ammonium; amine; glycol; ether; ethylene; e specific examples of such compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\* monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\*, and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene \*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type have been commercially marketed under the tradenames Dowanol, Carbitol, and Cellosolve. Another preferred type of nonaqueous, low-polarity organic solvent useful herein comprises the lower molecular weight polyethylene \*\*glycol\*\*s (PEGs). Such materials are those having molecular weights of at least about 150. PEGs of molecular weight ranging from about 200 to 600 are most preferred. Yet another preferred type of non-polar, nonaqueous solvent comprises lower molecular weight methyl esters. Such materials are those of the general formula: R.sup.1 --C(0)--OCH.sub.3 wherein R.sup.1 ranges from 1 to about 18. Examples of sui 6153577 classes:1 510/356 1 510/505 1 510/506 score: 436 keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd roxide; ethylene glycol; propylene glycol; propylene; pyrrolidone; amide; alkaline; buffered; sulfoxide; aqueous; a cid; ammonium; amine; glycol; ether; ethylene; itions are higher molecular weight nonionics, such as Neodol 45-11, which are similar \*\*ethylene\*\* oxide condensation products of higher fatty alcohols, with the higher fatty alcohol being of 14-15 carbon atoms and the number of \*\*ethylene\*\* oxide groups per mole being about 11. Such products have also been commercially marketed by Shell Chemical Company The alcohol alkoxylate component which is essentially utilized as part of the liquid diluent in the nonaqueous compositions herein will generally be present to the extent of from about 1% to 60% of the liquid phase composition. More preferably, the alcohol alkoxylate component will comprise about 5% to 40% of the liquid phase. Most preferably, the essentially utilized alcohol alkoxylate component will comprise from about 5% to 30% of the detergent composition liquid phase. Utilization of alcohol alkoxylate in these concentrations in the liquid phase corresponds to an alcohol alk branched or straight chain alkylene \*\*glycol\*\*s. Materials of this type include hexylene \*\*glycol\*\* (4-methyl-2,4-pentanediol), 1,6-hexanediol, 1,3-butylene \*\*glycol\*\* and 1,4-butylene \*\*glycol\*\*. Hexylene \*\*glycol\*\* is the most preferred. Another preferred type of nonaqueous, low-polarity solvent for use herein comprises the mono-, di-, tri-, or tetra- C.sub.2 -C.sub.3 alkylene \*\*glycol\*\*

mono C.sub.2 -C.sub.6 alkyl \*\*ether\*\*s. The specific examples of such

report09881552.txt

compounds include diethylene \*\*glycol\*\* monobutyl \*\*ether\*\*, \*\*tetraethyl\*\*ene \*\*glycol\*\*
monobutyl \*\*ether\*\*, dipropylene \*\*glycol\*\* monoethyl \*\*ether\*\*, and dipropylene

\*\*glycol\*\* monobutyl \*\*ether\*\*. Diethylene \*\*glycol\*\* monobutyl \*\*ether\*\* and dipropylene

\*\*glycol\*\* monobutyl \*\*ether\*\* are especially preferred. Compounds of the type
have been commercially marketed under the tradenames Dowanol, Carbitol,

Another preferred type of nonaqueous, low-polarity organic solvent useful herein comprises the lower molecular weight polyethylen

6448213

classes:1 510/357 1 510/424 1 510/426 1 510/428 1 568/458 1 560/76

score: 433

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;pyrrolidone;hydroxide;propylene glycol; propylene; pyrrolidone; amide; alkaline; buffered; sulfoxide; aqueous; acid; ammonium; amine; glycol; ether; ethylene;

	W			
_	W LAS	14	14	14
	C45 AS	2.4	2.4	2.4
	C45E1S	0.9	0.9	0.9
	C23E6.5	1.5	1.5	1.5
	C16 branched sulfate, sodiu		8.0	
	C17 branched sulfate, sodiu		8	.0 4.0
	C18 branched sulfate, sodiu			4.0
	QAS	1.5	1.5	1.5
	Zeolite A	26	26	26
	Carbonate	19.3	19.3	19.3
	Sulfate	5	5	5
	Perborate	1	1	1
	Polyacrylic Acid (MW = 4500		2	2
	Polyethylene **glycol** (MW		0.9	0.9 0.9
	Silicate	0.6	0.6	0.6
	Water		Balance -	-
-	AA	BB	CC	
	MBAS (14.5-15.5 ave. total carb			14
	C11.3 LAS	14	11	7.5
	QAS	1	1 2.5-3.5	1
	LMFAA	2.5-3.5 0.6-2	0.6-2	2.5-3.5 0.6-2
	C23E9 APA	0.6-2	0.6-2	0.6-2
	Citric Acid	3.0	3.0	3.0
	Fatty Acid (TPK or C12/14)	2.0	2.0	2.0
	Ethanol	3.4	3.4	3.4
	Propanediol	6.4	6.4	6.4
	Monoethanol **amine**	1.		
	NaOH	3.0	3.0	3.0
	Na toluene sulfonate	2.3	2.3	2.3
	Na formate	0.1	0.1	0.1
	Borax	2-2.5	2-2.5	2-2.5
	P			
-	EE FF			
	MBAS (14.5-15.5 ave. total carb	on) 13	10	7
	C11.3 LAS	7	10	13
	Any combination of:	1	1	1
	C25  AExS*Na (x = 1.8-2.5)			
	C25 AS (linear to high 2-alkyl)			
	C14-17 NaPS			
	C12-16 SAS			
	C18 1,4 disuifate			
	C12-16 MES		,	
	QAS	1	1	1
	LMFAA	3.5-5.5	3.5-5.5	3.5-5.5
	C23E9	4-6	4 – 6	4-6
	APA	0-1.5	0-1.5	0-1.5
	Citric Acid	1	1	1
	Fatty Acid (TPK or C12/14)	7.5	7.5	7.5
	Fatty Acid (Rapeseed)	3.1	3.1	3.1
	Ethanol	1.8	1.8	1.8
	Propanediol	9.4	9.4	9.4
	Monoethanol **amine**	6.	5 6.	5 6.5
	NaOH		D 0:	•

Page 22

----- 6228829

classes:1 510/357 1 510/424 1 510/426 1 510/428 1 560/76 1 568/458 1 568/882

score: 433

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;pyrrolidone;hydroxide;propylene
glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;
ethylene;

- 6306812

classes:1 510/310 1 8/111 1 8/137 1 134/252 1 134/42 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302 1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376

score: 432

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;am monium;amine;glycol;ether;ethylene;

olute ethanol (100 mL) and this solution is refluxed 1 hour. During that time, a tan solid formed which is separated from the mother liquor by vacuum filtration using Whatman #1 filter paper. The solid is dried under vacuum, overnight. Yield: 1.79 g, II, (15%). Fab Mass Spec. TG/G, MeOH) M.sup.+ 266 mu, 60%, MI.sup.+ 393 mu, 25%. Synthesis of 5,8 Dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane To a stirred solution of II, (1.78 g, 3.40 mmol) in ethanol (100 mL, 95%) is added sodium borohydride (3.78 g. 0.100 mmol). The reaction is stirred under nitrogen at RT for 4 days. 10% Hydrochloric acid is slowly added until the pH is 1-2 to decompose the unreacted NaBH.sub.4. Ethanol (70 mL) is then added. The solvent is removed by roto-evaporation under reduced pressure. The product is then dissolved in \*\*aqueous\*\* KOH (125 mL, 20%), resulting in a pH 14 solution. The product is then extracted with benzene (5.times.60 mL) and the combined

----- 6242406

classes:1 510/357 1 510/424 1 510/426 1 510/427 1 510/428 1 510/473

score: 432

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;propylene glycol;ethylene glycol;pyrrolidone;hyd
roxide;ethylene glycol;propylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;a
cid;ammonium;amine;glycol;ether;ethylene;

- Ze	eolite A	260	260	260
1	Methyl Cellulose	10	20	5
. 5	Sodium Carbonate	193	193	193
5	Sodium Sulfate	52	52	52
5	Sodium Perborate	10	10	10
1	Polyacrylic Acid (MW = 4500)	) 22	22	22
1	Polyethylene **glycol** (MW	= 9	9	9
4	4600)			
9	Sodium Silicate	6	6	6

EXAMPLE 15

The following laundry detergent compositions A to I are prepared in accord with the invention:

" T C C C		• •							
	A	В	С	D	E	F	G	Н	I
LAS	10	10	10	20	20	20	0	0	0
C45 AS	10	10	10	0	0	0	20	20	20
MBAE	1	2.5	5	1	2.5	5	1	2.5	5
Zeolite A	28	28	28	28	28	28	28	28	28
מאמ	2								

PAA 2 W X Y Z

MBAEx (x = 5-10; 0.5-5 4-6 10-15 20-25 14.6-15.5 ave. total carbon in alkyl group)

Any combination of: 21.5 19 5-15 1-6

C25 AExS\*Na (x = 1.8-2.5)

C25 AS (linear to high 2-alkyl)

C14-17 NaPS				
C12-16 SAS				
C18 1,4 disulfate				
C12-16 MES				
C11.3 LAS	5	1	10	15
LMFAA	2.5-5.5	2.5-5.5	0-3	0-3
Any combination of:	0-1.5	0-1.5	0-2	0-3
APA				
QAS				
C12-14 trimethyl				
**ammonium** halide				
DSDMAC			- <b>-</b>	4
Methyl Cellulose	3	3	1	5
Citric Acid	3	1	1	1
Fatty Acid (TPK, C12/	14 2	10.6	0-5	0-5
or Rapeseed)				
Ethanol	3.4	1.8	4	5.5
Propaneidol	6.4	9.4	6	

----- 6399557

classes:1 510/310 1 252/18633 1 510/220 1 510/221 1 510/224 1 510/302 1 510/303 1 510/304 1 510/311 1 510/312 1 510/313 1 510/314 1 510/372 1 510/376

score: 430

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;am monium;amine;glycol;ether;ethylene;

- he oil is taken up in absolute ethanol (100 mL) and this solution is refluxed 1 hour. During that time, a tan solid formed which is separated from the mother liquor by vacuum filtration using Whatman #1 filter paper. The solid is dried under vacuum, overnight. Yield: 1.79 g, II, (15%). Fab Mass Spec. TG/G, MeOH) M.sup.+ 266 mu, 60%, M.sup.+ 393 mu, 25%.

Synthesis of 5,8

Dimethyl-1,5,8,12-tetraazabicyclo.lambda.10.3.2]heptadecane
To a stirred solution of II, (1.78 g, 3.40 mmol) in ethanol (100 mL,95%) is
added sodium borohydride (3.78 g. 0.100 mmol). The reaction is stirred
under nitrogen at RT for 4 days. 10% Hydrochloric acid is slowly added
until the pH is 1-2 to decompose the unreacted NaBH.sub.4. Ethanol (70 mL)
is then added. The solvent is removed by roto-evaporation under reduced
pressure. The product is then dissolved in \*\*aqueous\*\* KOH (125 mL, 20%),
resulting in a pH 14 solution. The product is then extracted with benzen

4102823

classes:1 510/351 1 510/348 1 510/352 1 510/355 1 510/452 1 510/453 1 510/488

score: 428

keywords: alkaline; ph; alkaline; aqueous; acid; ethylene;

- erformance quality, physical characteristics and processability are prepared if, in the above-described Example IV composition, the sodium tripolyphosphate is replaced with an equivalent amount of potassium tripolyphosphate, potassium hexametaphosphate, tetrapotassium pyrophosphate or sodium pyrophosphate.

A composition of substantially similar performance quality, physical characteristics and processability is prepared if, in the above-described Example IV composition the hydrogenated fish oil fatty acid is replaced with an equivalent amount of tallow fatty acid or mixtures of tallow fatty acid and hydrogenated fish oil fatty acid containing about 18 carbon atoms.

WASH-WEAR TEST

Detergency performance of compositions of the instant invention is compared with that of a commercially-available built granular laundry detergent in a wash and wear test. The test employed is conducted in the following manner: Light-colored dress shi

----- 6306817

classes:1 510/535 1 510/357 1 510/424 1 510/426 1 510/428 Page 24 score: 427

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;hydroxide;ethylene glycol;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;ammonium;amine;glycol;ether;ethylene;

```
NOVO Industries A/S
MEA
            Monoethanolamine
PG
            Propanediol
EtOH
            Ethanol
NaOH
            Solution of sodium **hydroxide**
            Sodium toluene sulfonate
NaTS
Citric acid Anhydrous citric acid
CxyFA
            C.sub.1x --C.sub.1y fatty acid
            A C.sub.1x-1y branched primary alcohol condensed with an
CxyEz
            average of z moles of **ethylene** oxide
Carbonate
            Anhydrous sodium carbonate with a particle size
            between 200 .mu.m and 900 .mu.m
            Tri-sodium citrate dihydrate of activity 86.4% with a
Citrate
            particle size distribution between 425 .mu.m and 850 .mu.m
TFAA
            C16-18 alkyl N-methyl glucamide
LMFAA
            C12-14 alkyl N-methyl glucamide
            C8-C10 amido propyl dimethyl **amine**
APA
Fatty Acid C12-C14 fatty acid
(C12/14)
Fatty Acid Topped palm kernel fatty acid
(TPK)
Fatty Acid Rapeseed fatty acid
(RPS)
Borax
            Na tetraborate decahydr
       size in the range from 0.1 to 10 micrometers
NaSKS-6
            Crystalline layered silicate of formula .delta.-Na.sub.2
Si.sub.2 O.sub.5
Bicarbonate Anhydrous sodium bicarbonate with a particle size
            distribution between 400 .mu.m and 1200 .mu.m
            Amorphous Sodium Silicate (SiO.sub.2 :Na.sub.2 O; 2.0 ratio)
Silicate
Sulfate
            Anhydrous sodium sulfate
PAE
            ethoxylated **tetraethyl**ene pentamine
            ethoxylated polyethylene imine
PIE
PAEC
            methyl quaternized ethoxylated dihexylene triamine
MA/AA
            Copolymer of 1:4 maleic/acrylic acid, average
            molecular weight about 70,000.
CMC
            Sodium carboxymethyl cellulose
            Proteolytic enzyme of activity 4KNPU/g sold by
Protease
            NOVO Industries A/S under the tradename Savinase Cellulytic enzyme of activity 1000 CEVU/g sold by
Cellulase
            NOVO Industries A/S under the tradename
            Carezyme
Amylase
```

----- 6218351 classes:1 510/311 1 510/376 1 510/500 score: 425

keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzotriazole;hydroxide;ethylene glycol;propylene;pyrrolidone;amide;alkaline;buffered;sulfoxide;aqueous;acid;am monium;amine;glycol;ether;ethylene;

```
0.5
                                      0.5 :
                                                       1.00
Perfume
                                              2
                       0.75
Soil Release 0.50
                               0.75
                                       0.75
                                               0.50
                                                       0.75
Polymer.sup.5
Product of 2.5
                      10
                              5
                                      0.5
                                                      20
                                              1
Example.sup.6 ppm
                                                         ppm
                         ppm
                                 ppm
                                         ppm
                                                 ppm
                             to 100 to 100 to 100 to 100
            to 100
                      to 100
.sup.1 Di-(soft-tallowyloxyethyl) dimethyl **ammonium** chloride or
Distearyldimethylammonium chloride
.sup.2 Diethylenetriamine Pentaacetic acid(3) DC-2310, sold by Dow-Corning
.sup.3 DC-2310, sold by Dow-Corning
.sup.4 Kathon CG, sold by Rohm & Has
.sup.5 Copolymer of **propylene** terephthalate and **ethylene**oxide
.sup.6 Mn(Bcyclam)Cl.sub.2 as in Synthesis Example 1
                                             Page 25
```

```
EXAMPLE 27
     ##STR56##
Synthesis of 1,5,9,13-Tetraazatetracyclo[1.2.2.2.sup.5.9] heptadecane
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in
     acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,
               6387862
classes:1 510/311 1 510/376 1 510/500
score: 425
keywords: alkaline;ph;sulfoxide;amide;pyrrolidone;glycol;ethylene glycol;pyrrolidone;benzotriazole;benzot
riazole; hydroxide; ethylene glycol; propylene; pyrrolidone; amide; alkaline; buffered; sulfoxide; aqueous; acid; am
monium; amine; glycol; ether; ethylene;
     lease 0.50
                    0.75
                              0.75
                                       0.75
                                                0.50
                                                         0.75
    Polymer.sup.5
                   2.5
                            10
                                     5
                                              0.5
                                                               20
    Product of
    Example.sup.6 ppm
                             ppm
                                      ppm
                                              ppm
                                                       ppm
                                                                ppm
                   to 100 to 100 to 100 to 100 to 100
    .sup.1 Di-(soft-tallowyloxyethyl) dimethyl **ammonium** chloride or
     Distearyldimethylammonium chloride
    .sup.2 Diethylenetriamine Pentaacetic acid(3) DC-2310, sold by Dow-Corning
    .sup.3 DC-2310, sold by Dow-Corning
    .sup.4 Kathon CG, sold by Rohm & Has
    .sup.5 Copolymer of **propylene** terephthalate and **ethylene**oxide
     .sup.6 Mn(Bcyclam)Cl.sub.2 as in Synthesis Example 1
EXAMPLE 27
Dithiocyanato Manganese (II)
5,8 Dimethyl-1,5,8,12-tetraazabicyclo[10.3.2]heptadecane Synthesis
##STR54##
Synthesis of 1,5,9,13-Tetraazatetracyclo[11.2.2.2.sup.5,9] heptadecane
1,4,8,12-tetraazacyclopentadecane (4.00 g, 18.7 mmol) is suspended in
     acetonitrile (30 mL) under nitrogen and to this is added glyoxal (3.00 g,
     40% aqu
               6280527
classes:1 134/2 1 134/38
score: 420
keywords: alkaline;ph;glycol;glycol ether;ammonium hydroxide;ammonium hydroxide;hydroxide;glycol ether;te
traethyl; tetramethyl ammonium; tetramethyl; alkaline; aqueous; acid; ammonium; glycol; ether;
     clude polar,
     non-polar, hydrophilic, and hydrophobic types of pastes from stencil
     masks, typically Mo metal, electroform masks, and emulsion masks.
Tetramethyl **ammonium **hydroxide** (TMAH) is the preferred organic base for
   mask cleaning applications according to this invention but other related commonly known quaternary **ammonium **hydroxide**s that may also be used are:
     **tetraethyl ammonium **hydroxide**, tetrabutyl **ammonium **hydroxide**, trimethyl-2-hydroxyethyl **ammonium **hydroxide** (Choline), triethyl-2-hydroxy
ethyl **ammonium **hydroxide**, ethyltrimethyl **ammonium **hydroxide**, and the like. The **aqueous alkaline** cleaning solution comprising quaternary **ammonium**
     **hydroxide**s may optionally contain one or more corrosion inhibitor.
According to a preferred embodiment of this invention, a pressurized spray
     wash at between about 50 to about 200 psi with a heated **aqueous** solution
     of **tetramethyl ammonium **hydroxide** (TMAH) containing between about 0.3 and
     about 2.0
               5565136
classes:1 510/203 1 510/206 1 510/212 1 510/418
score: 414
keywords: glycol;glycol ether;propylene glycol;nmp;ammonium hydroxide;ammonium hydroxide;hydroxide;propyl
ene glycol;propylene;glycol ether;nmp;acid;ammonium;amine;glycol;ether;
      such as **ammonium **hydroxide** and
```

monoethanol \*\*amine\*\*, and acidic type such as formic acid, acetic acid and

oxalic acid are not necessary and preferably not included in the compositions of the present invention.

The water-based coating remover of the present invention is useful for removing various cured and uncured paints, varnishes, lacquers and other coatings or finishes, particularly coatings used on wood such as acrylic latex type enamel, polyurethane based clear protective coatings, oil-based alkyl resin, epoxy paint and varnish/lacquer.

The water-based coating remover of the present invention can be used for removing paint, lacquers, varnishes and other coatings or finishes from a variety of substrates, including paint brushes, without significant damage, such as etching, pitting or corrosion to such substrate. Examples of substrates on which the coating remover of the present invention can be used includes: wood; glass; ac

---- 5026933

classes:1 585/7 1 562/93 1 562/95 1 585/10 1 585/11 1 585/18 1 585/19 1 585/24 1 585/323 1 585/329 1 585/

455 1 585/467 1 585/533

score: 413

keywords: propylene;aqueous;acid;ammonium;amine;ethylene;

# - XVII

In accordance with this Example, benzene was alkylated with an olefin mixture over the dealuminized mordenite prepared in Example XVI. The olefin mixture was \*\*propylene\*\* tetramer (1.3 methyl branches per chain) prepared by oligomerizing \*\*propylene\*\* over a ZSM-23 catalyst having surface acid sites inactivated with 2,4,6-collidine and separating out the C.sub.12 fraction. This olefin mixture was percolated through activated alumina prior to use. To an oven-dried flask under N.sub.2 was added 10 ml perked C.sub.12 olefin (0.05 mol), 50 ml benzene (0.56 mol) and 0.6 g dealuminized mordenite. After 6 hr., GC showed about 10% reaction. Another 0.5 g catalyst was added and the mixture was refluxed 64 hrs. GC showed about 90% reaction. Another 0.25 g catalyst was added and the mixture was refluxed an additional 24 hr. The reaction was cooled, filtered, and evaporated to 10.15 g product, which still contained 4% unreacted olefin.

----- 5284989 classes:1 585/533 1 585/520

score: 409

keywords: propylene;aqueous;acid;ammonium;ethylene;

 hours. The resulting catalyst had an alpha value of 29 and a surface acidity less than 0.1.

Example 4

Surface Acidity Reduction with Collidine

A third sample of 7.7 g of the ZSM-23 catalyst from Example 1 having a silica/alumina ratio of 110:1 was treated with 0.059 g collidine/g of catalyst by contacting the catalyst with a dilute collidine-pentane solution containing 0.0454 g collidine which is an amount sufficient to poison 25% of acid sites present in the zeolite component of the catalyst. The collidine adsorbed very rapidly and completely to the catalyst. The catalyst was dried slowly and then purged with nitrogen.

Example 5

Oligomerization \*\*propylene\*\* with ZSM-23 at 225.degree. C.

The catalysts of Examples 1, 2, 3 and 4 were evaluated in a semi-batch autoclave for activity, product selectivity, and product structure with \*\*propylene\*\* feed on demand for five hours at 800 psig and 225.degree. C. Under the above conditions, unmodifie

- ethyls/C.sub.12 necessary for preparing desirable chemical intermediates. The oxalic acid treated catalyst did not require treatment with collidine to achieve the desired

product quality.

The determination of Branching Index is a useful and sensitive method practiced by those skilled in the arts to which the present invention applies and used to quantitatively assess the degree of linearity of a molecule or molecular mixture. The index is determined as follows: the C6 and C9 oligomers are first removed from the sample and the C12+ fraction is hydrogenated using Pd/charcoal catalyst in acetic acid. The

hydrogenated sample is extracted from the acetic acid into deuterochloroform and the 1H NMR spectrum determined. The branching index is defined as the ratio of the intensity (area) of the resonance due to CH3  $(0.7-1.0~{\rm ppm})$  divided by the sum of the intensities (areas) of the resonances due to CH3 (0.7.-1.0 ppm) and CH2 (1.1-

5334332

classes:1 510/175 1 134/2 1 134/40 1 510/108 1 510/245 1 510/254 1 510/434 1 510/489 1 510/492 1 510/505 score: 365

keywords: alkaline; amide; glycol; propylene glycol; ethylene glycol; hydroxide; ethylene glycol; propylene glyc ol;propylene;amide;alkaline;acid;ammonium;glycol;ethylene;

onoamines. The

alkanol group of the \*\*amine\*\*s preferably has from 1 to 5 carbon atoms.

Preferred alkanolamines suitable for use in the present invention can be

represented by the chemical formula R.sub.1 R.sub.2 --N--CH.sub.2 CH.sub.2 --O--R.sub.3 wherein R.sub.1 and R.sub.2 can be H, CH.sub.3, CH.sub.3

CH.sub.2 or CH.sub.2 CH.sub.2 OH and R.sub.3 is CH.sub.2 CH.sub.2 OH.

Examples of suitable alkanolamines include monoethanolamine,

diethanolamine, triethanolamine, tertiarybutyldiethanolamine isopropanolamine, 2-amino-1-propanol, 3-amino-1-propanol, isobutanolamine, 2-amino-2-ethoxyethanol, and 2-amino-2-ethoxy-propanol.

Polar solvents suitable for use in the stripping composition of the present invention include \*\*ethylene \*\*glycol\*\*, \*\*ethylene \*\*glycol\*\* alkyl \*\*ether\*\*, diethylene \*\*glycol\*\* alkyl \*\*ether\*\*, triethylene \*\*glycol\*\* alkyl \*\*ether\*\*, \*\*propylene \*\*glycol\*\*,

\*\*propylene \*\*glycol\*\* alkyl \*\*ether\*\*, dipropylene \*\*glycol\*\* alkyl \*\*ether\*\*, tripropylene \*\*glycol\*\* alkyl \*\*ether\*\*, N-su

like salts would not

therefore be suitable for use based upon the understood mechanism of ionic contamination in a microcircuit as caused by cleaning and set forth above.

As evident from the above formula, the carboxylic acid may be mono-, di-

or trisubstituted rather than tetra-substituted.

A preferred cleaning composition of the present invention includes 30% by

weight hydroxylamine, 25% by weight 2-amino-2-ethoxyethanol, 5% by weight 1,2-dihydroxybenzene and 50% by weight of water.

The stripping compositions of the present composition are effective in removing a wide range of positive photoresists but are particularly useful in removing photoresists commonly consisting of an ortho-naphthoquinone diazide sulfonic acid ester or \*\*amide\*\* sensitizer with novolak-type binders or resins. Examples of commercially available photoresist compositions which the stripping compositions of the present invention effectively

remove from a su

classes:1 560/99 1 508/496 1 524/314 1 560/76 1 560/97 1 560/190 1 560/202 1 560/204 1 568/909

score: 221

keywords: weak; propylene; acid; ethylene;

anhydride (22.2 g, 0.15 mole), tridecanol (60.11 g, 0.3 mole, containing 1.4 branching methyl groups), and p-toluene sulfonic acid (0.1 g). The reaction is heated under reflux for 20 hours. Three milliliters of water is collected. Removing the solvent under a reduced pressure gave 78.2 g (95%) of ditridecylphthalate, a yellow liquid whose viscosity at

40.degree. C. is 43.5 centistokes.

In comparison, isotridecyl alcohol, which contains 3-4 branching methyl groups, and phthalic anhydride give a product whose viscosity at 40.degree. C. is 83 centistokes. These data show that the more linear alcohol gives a product with improved low temperature properties. Also, one skilled in the art recognizes that more linear alcohols decrease the volatility of esters such as this phthalate. These properties are useful for plasticizing PVC and other plastics. Ditridecylphthalate is also useful as a high temperature lubricant, and finds

ant properties for some

plasticizers.

Procedure for Ethoxylating Tridecanols. This procedure follows those by Satkowski and Hsu, and a Shell Chemical Company Bulletin, see Ind. Eng. Chem.; Satkowski, W. B., Hsu, C. G., "Polyoxyethylation of Alcohol", 1957,

49, 1975, and describes synthesizing a tridecanol-ethoxylate containing nine \*\*ethylene\*\* oxide residues. A 500 mL autoclave is charged with tridecanol [50 g (approximately 60 mL), 0.25 mole] and potassium \*\*hydroxide\*\* (0.15 g, 0.00268 mole), and heated at 135.degree. C. under vacuum (15 mm Hg or lower) for one hour. This removes water. Nitrogen gas is added until the vessel's pressure is 45 psig. Liquid \*\*ethylene\*\* oxide [99 g (113 mL), 2.25 mole) is fed on pressure demand from a calibrated, nitrogen gas blanketed Jurgenson gauge which is fitted with a check valve. \*\*ethylene\*\* oxide flowed once the reactor's pressure dropped below the pressure of the nitrogen gas blanket. Temperatu

Orleans, May 1981. Also, Shell Chemical Co. Technical Bulletin SC:580.82. The above-references are hereby incorporated by reference. An acetylation reagent is prepared by slowly adding acetic anhydride (120 mL) with stirring to a solution of p-toluene sulfonic acid monohydrate (14.4 g) and ethyl acetate (360 mL). This reagent is stored in a tightly sealed bottle shielded from light. A 100 mL flask, equipped with a magnetic spin bar and drying tube, is purged with nitrogen gas. Four grams of alcohol-ethoxylate is dissolved in 4 mL of toluene and transferred to the flask along with 12 mL of acetylation reagent. This solution is heated at 50.degree. C. for 30 minutes, transferred to a separatory funnel along with 40 mL of toluene, and washed three times: 100 mL of 30 wt % brine; 100 mL of bicarbonate-brine (50 g sodium bicarbonate, 250 g sodium chloride, 1750 g water); 100 mL of 30 wt % brine. The toluene solution is dried